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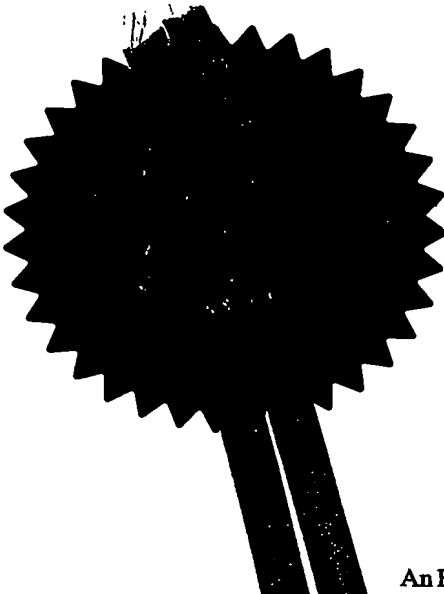
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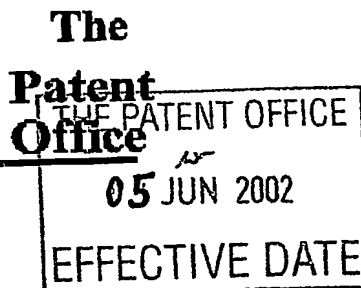
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2002CH004

2. Patent application number

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0212691.0

ACTH002 E733105-1 002992

7700 0.00-0212691.0

3. Full name, address and postcode of the or of each applicant (underline all surnames)

CLARIANT INTERNATIONAL LTD
Rothausstrasse 61
CH-4132 Muttenz

Patents ADP number (if you know it)

06971634001 ✓

If the applicant is a corporate body, give the country/state of its incorporation

Switzerland

4. Title of the invention

COMPOSITION FOR DYEING POLYESTER
TEXTILE MATERIALS

5. Name of your agent (if you have one)

CLARIANT UK LTD.

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

Attn. Mr. Stephen Parkinson
Calverley Lane
Horsforth/Leeds, LS18 4RP

Patents ADP number (if you know it)

7156086001 ✓

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Number of earlier application

Date of filing
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Yes

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Description	8
Claim(s)	4
Abstract	1
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Priority documents

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Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

1

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

11. I/We request the grant of a patent on the basis of this application.

Signature **Clariant International Ltd** Date **June 4, 2002**

Jörg Hermann
Dr. Jörg Hermann
Patent Attorney

C. Heusler
Christel Heusler
Patent Administrator

12. Name and daytime telephone number of person to contact in the United Kingdom

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1 DUPLICATE

Composition for Dyeing Polyester Textile Materials

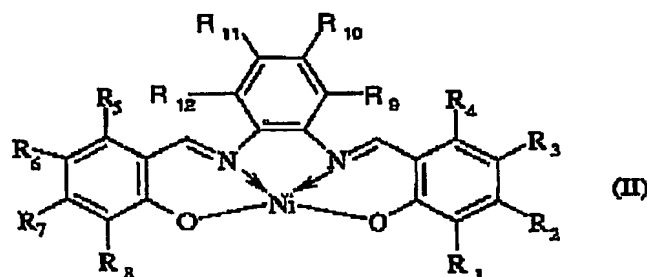
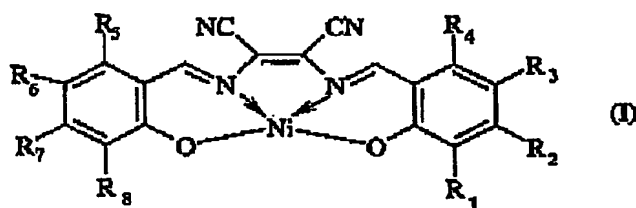
The invention relates to the use of a transition metal compound for dyeing a fibre fabric or a fibre material comprising polyester fibres for improving the light fastness of the dyeings.

The problem to be solved of the present invention is to improve the fastness to light of dyed polyester material.

10 It has been found, that the use of mixtures of at least one transition metal compound for dyeing a fibre fabric or a fibre material comprising polyester fibres improves the light fastness of the dyeings.

By preference, the transition metal complex comprises Ni, Co, Cr or Cu (Nickel, Cobalt, Chromium or Copper). The most preferred transition metal is Nickel (Ni).

For example, the mixture comprises at least one of the transition metal compound according to formula (I) or formula (II)



20

wherein $R_1 - R_{12}$ independently from each other signify H, halogen, $-\text{NO}_2$, $-\text{CN}$, $-\text{OH}$, $-\text{COOH}$, $-\text{CH}_3$, $-\text{NH}_2$ or NHCH_3 .

Preferably, transition metal compounds or mixtures of transition metal compounds are
5 used wherein $R_1 - R_{12}$ independently from each other signify H, $-\text{Cl}$, $-\text{COOH}$, or $-\text{CN}$.

The transition metal compound of formula (I), wherein all $R_1 - R_8$ are H, is known as
C.I. Solvent Brown 53 (Polysynthren® Braun R, Polysynthren® is a registered
Trademark of CLARIANT) or a transition metal compound of formula (II) wherein all
10 $R_1 - R_{12}$ are H, which is known as C.I. Pigment Orange 70 (Polysynthren® Braun 3RL,
Polysynthren® is a registered Trademark of CLARIANT).

Therefore this invention comprises the use of at least one transition metal compound for
improving the light fastness of dyed polyester material. Preferred transition metal
15 compound comprises Ni, Co, Cr or Cu (Nickel, Cobalt, Chromium or Copper); more
preferred transition metal compound comprises Nickel (Ni); and the most preferred
transition metal compound is either a compound according to formula (I) or formula (II)

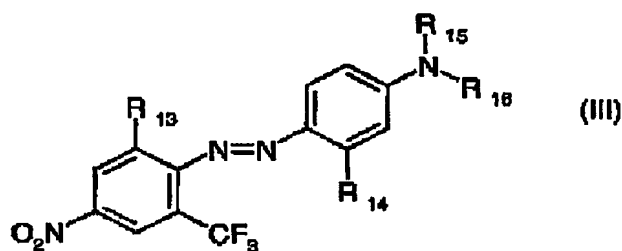
The transition metal compounds may be used together with other dyes suitable for
20 dyeing polyester materials. Usually polyester materials are dyed using disperse dyes.

In a more preferred embodiment the the transition metal complex comprises Ni, Co, Cr
or Cu (Nickel, Cobalt, Chromium or Copper). The most preferred transition metal is
Nickel (Ni). By preference these transition metal compounds or pigments are used in
25 mixtures together with disperse dyes. A suitable transition metal compound according
to formula (I) or formula (II) is either C.I. Solvent Brown 53 or C.I. Pigment Orange 70.

In the most preferred embodiment the transition metal compounds or the pigments
have the formula (I) or formula (II) and are used in mixtures together with disperse
30 dyes.

Preferred disperse dyes are for example at least one of the following disperse dyes: C.I.
Disperse Yellow 42, C.I. Disperse Yellow 72, C.I. Disperse Yellow 86, C.I. Disperse

Yellow 54, C.I. Disperse Yellow 64, C.I. Solvent Yellow 163, C.I. Disperse Red 60, C.I. Disperse Red 86, C.I. Disperse Red 91, C.I. Disperse Red 167, C.I. Disperse Red 167.1, C.I. Disperse Red 202, C.I. Disperse Red 302, C.I. Disperse Red 273, C.I. Disperse Red 279, C.I. Disperse Red 271, C.I. Solvent Red 135, C.I. Disperse Violet 27, C.I. Disperse Violet 57, C.I. Disperse Blue 56, C.I. Disperse Blue 77, C.I. Disperse Blue 54, C.I. Disperse Blue 27, C.I. Disperse Blue 55, C.I. Disperse Blue 60, C.I. Disperse Blue 87, C.I. Disperse Orange 30, C.I. Disperse Orange 41, C.I. Disperse Orange 29, structures according to formula (III)



wherein

R_{13} signifies -Br, -Cl, or -CN;

R_{14} signifies -H, -CH₃, -NHCOCH₃;

R_{15} signifies a unsubstituted ethyl group or ethyl group

which is substituted by -CN, -acyloxy;

R_{16} signifies a unsubstituted ethyl group or ethyl group

which is substituted by -CN, -acyloxy;

or mixtures thereof.

Dyes according to the formula (III) are known for example from EP1085055 A1, CH468444, US2941992, US3407189, FR1291988, US2891942, DE2364205 and JP49030417 A. Preferred examples are shown in EP1085055 A1 on page 2, line 37 to page 4, line 23 and in the examples 1 to 20 of EP1085055 A1.

A further aspect of the present invention is a mixture of at least one transition metal compound comprising Nickel (Ni) and at least one disperse dye. In a preferred embodiment the Ni-compound is of formula (I) or formula (II).

The transition metal complexes according to the invention can be used in any ratio with disperse dyes. For example the mixture comprises 2% to 99% of the transition metal complexes, preferably from 50% to 97%.

- 5 According to the invention, the pigments of formula (I) or formula (II) as well as mixtures comprising the pigments of formula (I) or formula (II) are used for dyeing and printing semisynthetic and, preferably, synthetic hydrophobic fiber materials, especially textile materials. Textile materials consisting of blended fabrics containing such semisynthetic hydrophobic fiber materials can also be dyed or printed by means of the
10 dyes of this invention.

Suitable semisynthetic textile materials are mainly cellulose-2½ acetate, cellulose triacetate polyamides and high molecular weight polyesters as well as mixtures thereof with cellulose.

15

- Synthetic hydrophobic textile materials consist mainly of linear aromatic polyester, for example of those consisting of terephthalic acid and glycols, in particular ethylene glycol or condensate of terephthalic acid and 1,4-bis(hydroxymethyl)cyclohexane; of polycarbonates, e.g. those consisting of alpha,alpha-dimethyl-4,4'-dihydroxydiphenyl-
20 methane and phosgene, and of fibers based on polyvinyl chloride and polyamide.

- The hydrophobic synthetic materials can be in the form of sheet-like or thread-like structures, and can be processed, for example, to yarns or woven, knitted or looped textile fabrics. The mixtures according to the invention are also suitable for dyeing
25 hydrophobic synthetic material in the form of micro fibers.

- Dyeing can be carried out by known methods. Additives usual for dyeing with or dyeing with disperse dyes can be added to the bath (e.g. dispersing agents, preferably anionic dispersing agents, NaCl and/or Glauber salt)

30

Preferably dyeing with the disperse dyes (e.g. for the polyester fibres of a mixed fibre substrate) is performed at pH of 3-9 more preferably 4-6, most preferably 4-5.5 and at a temperature of 125 DEG-135 DEG C. for 15-45 minutes.

After dyeing in a process according to the invention, conventional washing and drying steps may be employed.

- 5 The textile material dyed using a mixture comprising a transition metal complex and a disperse dye provides dyed textile materials having good light fastness. This dyed textile material is suitable for example as upholstery material in car manufacturing or any other application wherever improved light fastness is desired for example for an article of clothing or for a sun blind or textiles for outdoor furniture.

10

APPLICATION EXAMPLE

- 17.5 parts of the pigment and disperse dye mixture (96% C.I. Solvent Brown 53 and 4%
15 C.I. Disperse Red 86; this is the dye mixture from example 23a of table 1) with 32.5 parts of a commercial dispersing agent based on lignin sulphonates, and pulverized to a powder. 1.2 parts of this dye preparation are added to 2000 parts of demineralized water of 70°C, which contains 40 parts of ammonium sulfate; the pH value of the dye bath is set at 5 with 85% formic acid. 100 parts of washed polyester fiber fabric are placed in
20 this dye bath, the container is closed, heated to 130°C over the course of 20 minutes, and dyeing continues for a further 60 minutes at this temperature. After cooling, the polyester fiber fabric is removed from the dye bath, rinsed, soaped and cleansed by reduction with sodium hydrosulphite in the usual way. After thermo-fixation (180°C, 30 min), a brownish pink dyeing is obtained with very good all-round fastness, especially
25 fastness to light and sublimation, in particular excellent wet fastness.

- The dyed swatches were exposed to light by the fakra standard test. FAKRA is a test according to ISO 105/B02: Exposure in Xenotest 450 equipped with a Xenon arc source; black standard temperature 45 °C, relative humidity 45 ± 5 %. The number of
30 cycles is indicated in tables as times this cycle: 2 cycles fakra means two cycles of this Fakra test; in DIN 75202/H: one cycle FAKRA is 54 hours exposure.

Afterwards the adjacent fabrics are assessed by the Grey Scale Change ISO A03.

- This 5-step Grey Scale consists of 5 pairs of swatches of grey and white cloth which illustrate the perceived color differences corresponding to fastness ratings 5, 4-5, 4, etc. The fastness rating is that number of the Grey Scale which has a perceived color difference equal in magnitude to the perceived color difference between the original adjacent fabric and the treated adjacent fabric. An improvement of a value by 1 signifies an improvement of 20%

The results are summarized in table 1, table 2, table 3 and table 4.

- 10 In tables the amount of the dyes is given in percent. Eventually an additional UV Absorber is applied, for example Fadex ECS liq (registered Trademark of Clariant AG, Muttenz, Switzerland). The amount of the UV absorber is given in percent of the weight of the fabric treated. The results of the fakra test is given as explained above

15 Table 1

Dye	23a	24a	25a	26a	27a	28a
C.I. Solvent Yellow 163	0	0	9	0	0	33
C.I. Solvent Brown 53	96	96	90.9	83	83	67
C.I. Disperse Red 86	4	0	0	17	0.0	0
C.I. Disperse Blue 77	0	4	0	0	17	0
Fadex ECS liq.	2	2	2	2	2	2
2 cycles Fakra	4.5	4.6	4.7	4.8	4.9	4.9
4 cycles Fakra	4.3	4.3	4.5	4.6	4.7	4.7
6 cycles Fakra	4.1	4.2	4.5	4.5	4.6	4.6
8 cycles Fakra	4.0	4.1	4.3	4.4	4.5	4.7
10 cycles Fakra	3.7	3.9	4.2	4.1	4.3	4.6

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Table 2

Dye	29a	30a	31a	32a	33a	34a
C.I. Solvent Yellow 163	0	0	9	0	0	33
C.I. Pigment Orange 70	96	96	91	83	83	67
C.I. Disperse Red 86	4	0	0	17	0	0
C.I. Disperse Blue 77	0	4	0	0	17	0
Fadex ECS liq.	2	2	2	2	2	2
2 cycles Fakra	4.4	4.5	4.6	4.8	4.8	4.8
4 cycles Fakra	4.1	4.2	4.3	4.4	4.6	4.7
6 cycles Fakra	3.8	3.9	4.1	4.2	4.3	4.5
8 cycles Fakra	3.6	3.8	4.1	4.0	4.3	4.4
10 cycles Fakra	3.4	3.6	3.8	3.8	4.2	4.2

5

Tabel 3 (The examples 1a, 2a are 1b are comparative examples.)

dye	1a	2a	3a
C.I. Disperse Yellow 42	57	0	0
C.I. Solvent Yellow 163	0	45	22
Mixture of C.I. Disperse Red 86 and C.I. Solvent Red 135	0	24	12
C.I. Disperse Red 86	21	0	0
C.I. Solvent Brown 53	0	0	47
C.I. Disperse Blue 77	15	31	0
C.I. Disperse Blue 60	7	0	18
Fadex ECS liq.	2	2	2
7 cycles Fakra	2.7	2.5	3.5

10

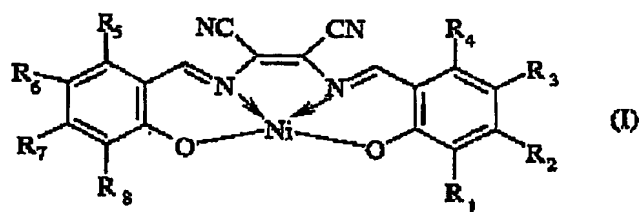
Table 4 (The examples 2b, 4b, and 5b are comparative examples)

dye	1b	2b	3b	4b	5b
C.I. Disperse Yellow 42	49	0	0	44	0
C.I. Solvent Yellow 163	0	33	0	0	30
Mixture of C.I. Disperse Red 86 and C.I. Solvent Red 135	0	24	2	0	24
C.I. Disperse Red 86	25	0	0	25	0
C.I. Solvent Brown 53	0	0	13	0	0
C.I. Pigment Orange 70	0	0	53	0	0
C.I. Disperse Blue 77	27	43	31	31	45
C.I. Disperse Blue 60	8	0	0	9	0
Fadex ECS liq.	2	2	2	2	2
7 cycles Fakra	2.3	2.7	3.5	2.7	2.5

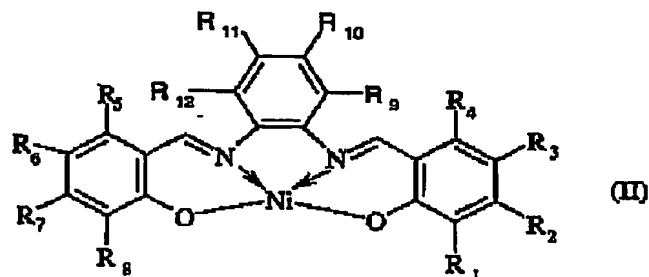
The examples 1a, 2a, 1b, 2b, 4b, and 5b are comparative examples. In table 3 green shades are realized and example 1a (comparative) has the same shade as example 3a. In table 4 dark green shades are realized.

CLAIMS

1. Use of at least one transition metal compound for improving the light fastness of dyed polyester material.
2. Use according to claim 1 characterized in that the transition metal compound comprises Ni, Co, Cr or Cu (Nickel, Cobalt, Chromium or Copper).
3. Use according to claim 2 characterized in that the transition metal compound comprises Nickel (Ni).
4. Use according to claim 3 characterized in that the transition metal compound is a compound according to formula (I) or formula (II)



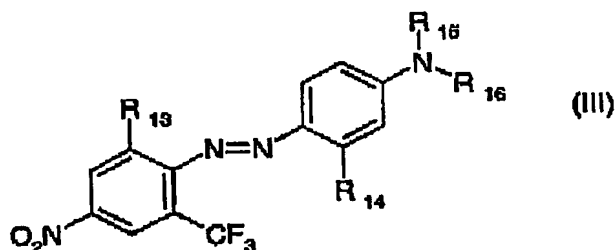
or



wherein $R_1 - R_{12}$ independently from each other signify H, halogen, $-\text{NO}_2$, $-\text{CN}$, $-\text{OH}$, $-\text{COOH}$, $-\text{CH}_3$, $-\text{NH}_2$ or NHCH_3 .

5. Use according to claim 5 characterized in that the transition metal compound is C.I. Solvent Brown 53 or C.I. Pigment Orange 70.

6. Use according to any of the claims 1 to 5 characterized in that the transition metal compound is used in a mixture comprising at least one of the following dyes: C.I. Disperse Yellow 42, C.I. Disperse Yellow 72, C.I. Disperse Yellow 86, C.I. Disperse Yellow 54, C.I. Disperse Yellow 64, C.I. Solvent Yellow 163, C.I. Disperse Red 60, C.I. Disperse Red 86, C.I. Disperse Red 91, C.I. Disperse Red 167, C.I. Disperse Red 167.1, C.I. Disperse Red 202, C.I. Disperse Red 302, C.I. Disperse Red 273, C.I. Disperse Red 279, C.I. Disperse Red 271, C.I. Solvent Red 135, C.I. Disperse Violet 27, C.I. Disperse Violet 57, C.I. Disperse Blue 56, C.I. Disperse Blue 77, C.I. Disperse Blue 54, C.I. Disperse Blue 27, C.I. Disperse Blue 55, C.I. Disperse Blue 60, C.I. Disperse Blue 87, C.I. Disperse Orange 30, C.I. Disperse Orange 41, C.I. Disperse Orange 29, structures according to formula (III)



wherein

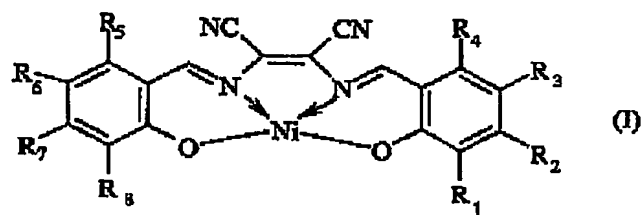
- R_{13} signifies -Br, -Cl, or -CN;
 R_{14} signifies -H, -CH₃, -NHCOCH₃;
 R_{15} signifies a unsubstituted ethyl group or ethyl group which is substituted by -CN, -acyloxy;
 R_{16} signifies a unsubstituted ethyl group or ethyl group which is substituted by -CN, -acyloxy;

or mixtures thereof.

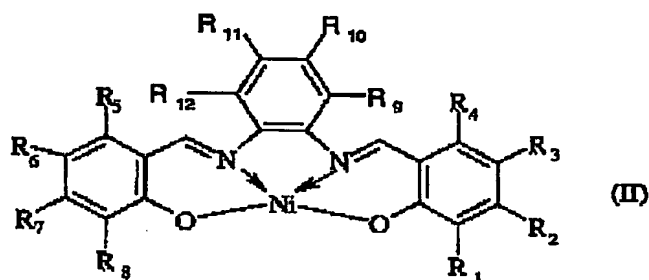
7. Mixture comprising at least one transition metal compound which comprises Nickel (Ni) and at least one disperse dye.

8. Mixture according to claim 7 characterized in that the at least one transition metal compound is a transition metal compound according to formula (I) or according to formula (II)

5



or

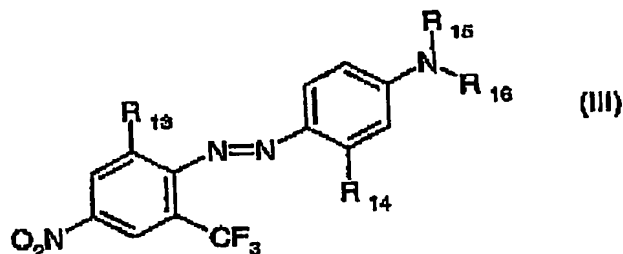


- 10 wherein R₁ - R₁₂ independently from each other signify H, halogen, -NO₂, -CN, -OH, -COOH, -CH₃, -NH₂ or NHCH₃

and

- 15 the at least one disperse dye is at least one of the following dyes: C.I. Disperse Yellow 42, C.I. Disperse Yellow 72, C.I. Disperse Yellow 86, C.I. Disperse Yellow 54, C.I. Disperse Yellow 64, C.I. Solvent Yellow 163, C.I. Disperse Red 60, C.I. Disperse Red 86, C.I. Disperse Red 91, C.I. Disperse Red 167, C.I. Disperse Red 167.1, C.I. Disperse Red 202, C.I. Disperse Red 302, C.I. Disperse Red 273, C.I. Disperse Red 279, C.I. Disperse Red 271, C.I. Solvent Red 135, C.I. Disperse Violet 27, C.I. Disperse Violet 57, C.I. Disperse Blue 56, C.I. Disperse Blue 77, C.I. Disperse Blue 54, C.I. Disperse Blue 27, C.I. Disperse Blue 55, C.I. Disperse Blue 60, C.I. Disperse Blue 87, C.I. Disperse Orange 30,
- 20

C.I. Disperse Orange 41, C.I. Disperse Orange 29, structures according to formula (III)



5 wherein

R_{13} signifies -Br, -Cl, or -CN;

R_{14} signifies -H, -CH₃, -NHCOCH₃;

R_{15} signifies a unsubstituted ethyl group or ethyl group which is
 substituted by -CN, -acyloxy;

10 R_{16} signifies a unsubstituted ethyl group or ethyl group which is
 substituted by -CN, -acyloxy;

or mixtures thereof.

15 9. Textile material dyed with a mixture according to Claim 8.

10. Use of a textile as claimed in Claim 9 as automobile upholstery or as an article
 of clothing or as a sun blind or textiles for out door furnitures.

ABSTRACT

Use of at least one transition metal compound for improving the light fastness of dyed polyester material. The transition metal compound may be used together with disperse dyes.

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